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A11102128969

/Bureau of Standards journal of research
QC1 .U52 V10;1933 C.1 NBS-PUB-C 1928

THE THERMAL EXPANSION OF REFRACTORIES TO 1,800° C.

By R. A. Heindl

ABSTRACT

The linear thermal expansions of the following 36 materials were measured: African chrome sand; Cuban, Grecian, Friable African, Rhodesian Imperial, Indian and Turkish chrome ores; Austrian, Californian, and electrically fused magnesites; a periclase brick; a spinel brick; two types of fire-clay bricks and two of fire clays; Kentucky, Tennessee, and English ball clays; Georgia kaolin and and English china clay; an 80 percent alumina brick; artificial corundum, diaspore, bauxite; five mullites, each of which was prepared from different raw materials; two zircon bricks and a furnaced zirconium silicate; silicon carbide, a silica brick, and an insulating brick. Data are also given on artificial graphite which had been preheated several times to 1,800° C. Measurements were made below 1,000° C. in both an oxidizing and a reducing atmosphere. Above, 1,000° C. the materials were tested in a reducing atmosphere only. When the refractoriness of the materials permitted they were tested up to 1,800° C. Petrographic analyses of the materials were made before and after the several heat treatments.

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I. INTRODUCTION

The need for information relative to the expansion of refractory materials, up to and beyond the temperature range in which they are now used, is evidenced by an increasing number of reports [1] dealing with the subject. Norton [2] gave reasons regarding the value of such

[1] F. H. Norton, The Thermal Expansion of Refractories, J.Am.Cer. Soc., vol. 8, no. 12, pp. 799–815, 1925.
K. Becker, X-ray Method of Determining Coefficient of Expansion at High Temperatures, Z. Physik, vol. 40, pp. 37–41, 1926.
W. Miehr, J. Kratzert, and H. Immke, The Importance of Thermal Expansion in the Valuation of Refractory Material, Tonin. Ztg., vol. 51 (26), pp. 417–422, 1927.
Hans Hirsch and Max Pulfrich, Expansion Measurements at high Temperatures, Tonin. Ztg., vol. 52 (36), pp. 712–713, 1928.
K. Endell and W. Steger, The Measurement of the Thermal Expansion of Refractory Materials to 1,600° C., Archiv. Eisenhüttenives, vol. 1 (11), 1928; Feuerfest vol. 5 (1), pp. 3–4, 1929.
Willi M. Cohn, Expansion Measurements from 20° to 1,400° C., Keramos, vol. 9 (7), pp. 215–218, 1930.
H. Reich, A New Apparatus for Measuring the Thermal Expansion of Refractory Materials in the Temperature Range 0° to 1,700° C., Berichte Deut. Ker. Gesell., vol. 13 (4), pp. 157–166, 1932.

[2] F. H. Norton, The Thermal Expansion of Refractories, J.Am.Cer. Soc., vol. 8, no. 12, pp. 799–815, 1925.

information. It may also be pointed out that it is not at all unusual for refractories to be used at temperatures considerably higher than those at which they had been heated during the manufacturing process. In such cases physical and chemical changes not completed during the manufacturing stage continue to progress. In many cases the linear thermal expansion is affected by such additional heating which in turn may greatly affect the stresses set up in the refractory during use.

The present report deals not only with the expansion of the well-known types of refractory materials to temperatures sufficiently high to cause many of them to soften or deform but also with the effect on the expansion when the materials are heated to a higher temperature than that at which they had been heated preliminary to the first test.

II. MATERIALS AND SPECIMENS

Seven chrome ores, four magnesites, five mullites, two fire-clay bricks, two fire clays, three ball clays, two kaolins, three zircons, and one each of spinel, silicon carbide, artificial corundum, diaspore, bauxite, 80 percent alumina fire brick, silica brick, and insulating brick were included in this investigation. A large proportion of these materials were furnished by manufacturers [3] and the others were available in the bureau laboratories.

Test specimens approximately 5⅞ inches long and ⅞ inch square were cut from commercial bricks if available, but if bricks were not available specimens of similar dimensions were prepared from raw materials which had been ground to pass a 40-mesh sieve. The specimens were prepared by adding sufficient water or gum tragacanth to each ground material so it could be readily molded. After removing the damp specimens from the mold they were dried, heated, and cut to the required dimensions. The specimens cut from the bricks were tested without further treatment whereas most of those prepared from the raw material were tested after having been heated at 1,400° C. for 5 hours and cooled with the furnace in about 36 hours.

III. APPARATUS

1. DESCRIPTION

A furnace of the high frequency induction type shown in figure 1 was used for determining the expansion of the various materials up to 1,800° C. The heat is generated through the medium of a cylindrical graphite muffle 12 inches long, 6 inches in diameter, and having a wall ½ inch thick. The gradual oxidation of the graphite muffle was the cause of a reducing atmosphere within the furnace. The converter for controlling the power input to the furnace is shown on the right in the figure. An Ames micrometer dial graduated in ten thousandth inch was supported independently of the furnace by a framework, the legs of which were fused quartz tubes.

The set-up of the specimen is indicated by the sketch in figure 2. This set-up formed a continuous system which also could expand and contract independently of the furnace. Tungsten plates ⅞ inch

[3] E. J. Lavino & Co., Philadelphia, Pa.; Harbison-Walker Refractories Co., Pittsburgh, Pa.; Norton Co., Worcester, Mass.; A. P. Green Fire Brick Co., Mexico, Mo.; Champion Spark Plug Co., Detroit, Mich.; Titanium Alloy Manufacturing Co., Niagara Falls, N.Y.; Babcock & Wilcox Co., New York, N.Y.

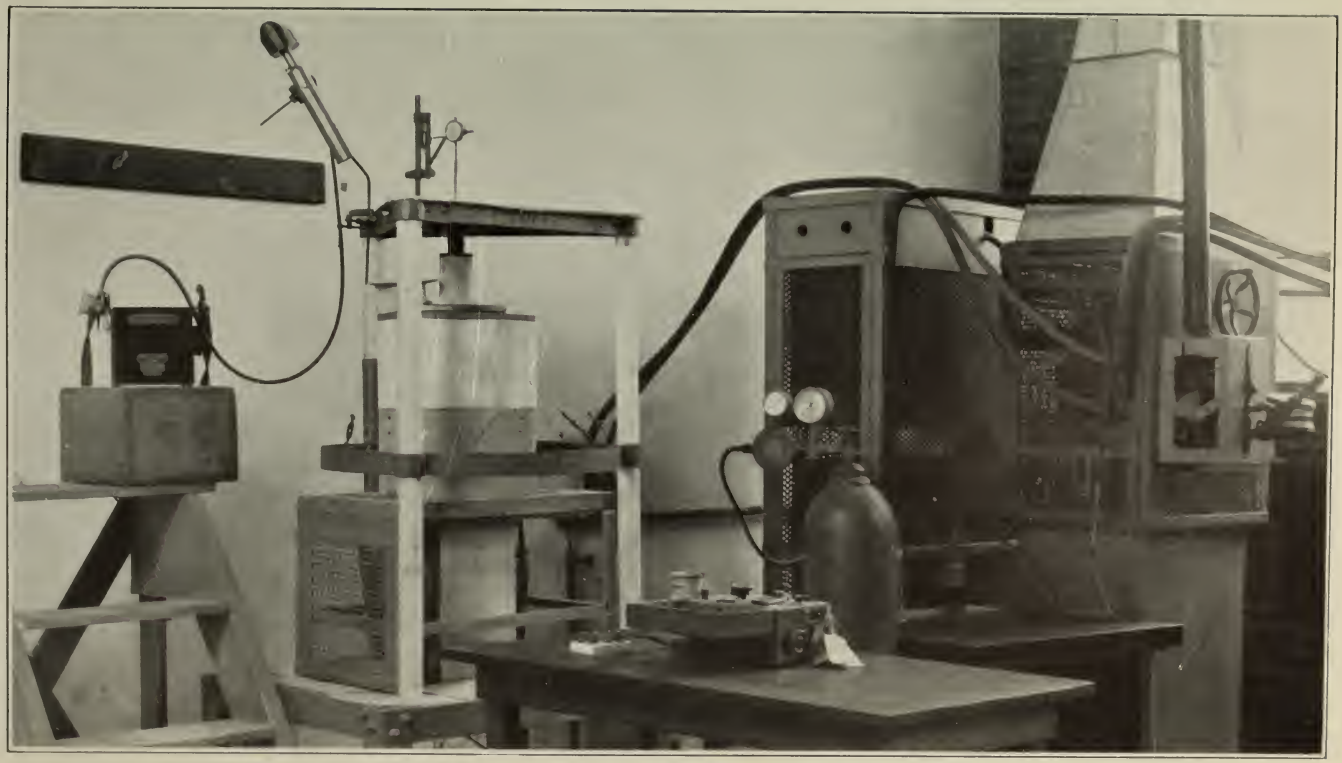

FIGURE 1.—*Apparatus used for obtaining linear thermal expansion measurements to 1,800° C.*

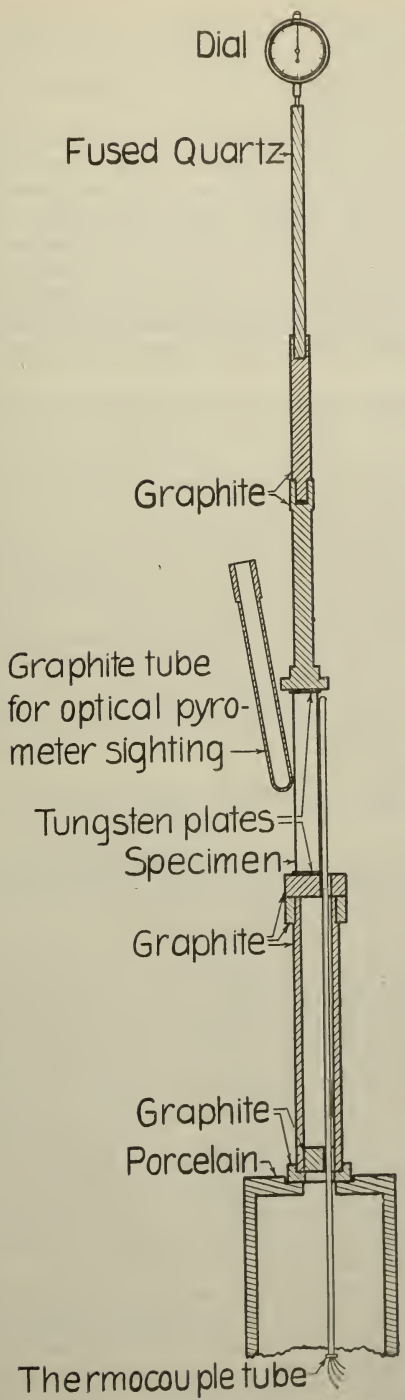

FIGURE 2.—*Showing the specimen, specimen support, and the system transmitting the expansion to the micrometer dial.*

The fused quartz rod does not extend into the furnace.

square and 0.015 inch thick were placed between the test specimen and the graphite pieces. The specimen was placed within a muffle, inside diameter 2¼ inches, prepared from zirconium silicate. The weight of the pieces resting on top of a specimen and making contact with the dial was 4 ounces.

Two platinum to platinum-rhodium thermocouples were placed alongside the specimen, one approximately three fourths inch below the top end and the other the same distance above the bottom end. These couples could be readily removed during a test through the lower end of the furnace without disturbing the set-up. The optical pyrometer shown in figure 1 was fixed in position, preceding the test, so that it sighted on the bottom of the closed end graphite tube indicated in figure 2. This tube was placed near the middle of the specimen and as close to it as possible without actually touching it.

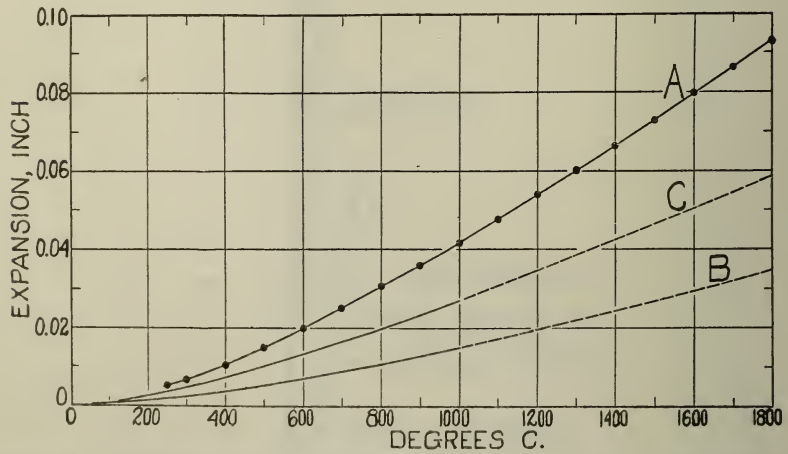

FIGURE 3.—*Calibration curve C for the apparatus when samples approximately 5⅞ inches long were tested and curves A and B involved in its derivation.*

2. CALIBRATION

The movements indicated by the micrometer dial during an expansion test represented the difference between the total expansion of the built-up column composed of the various parts shown in figure 2 and the vertical expansion of the outer framework and dial support shown in figure 1. The first step in the calibration was to make several tests with a bar of artificial graphite, 5.737 inches long and 1 inch in diameter, as the specimen. In each of these tests the temperatures were increased to a maximum of 1,800° C. at the same rate and the dial readings were recorded in the same manner as in the later tests. After preliminary heatings the dial readings for like temperatures in different tests were nearly the same and when plotted were found to lie along the smooth curve *A* in figure 3. This established the fact that the expansions of the entire measuring system were practically the same in repeated tests with the same specimen and set-up. During the course of the investigation several additional tests were made with the graphite bar as the specimen and in these, the readings, beginning at approximately 250° C. did not deviate more than 2.0 percent from curve *A*, figure 3.

Repeated readings over a temperature range of 20° to 1,000° C. were then made with a bar (5.852 inches long) of fused quartz, the thermal expansion of which was known. In these tests the rate of increase of temperature was the same as in other tests. Dial readings in the different tests of fused quartz checked, which confirmed the indications of the previous tests that the effects of expansions of supports for the specimen and the dial did not vary significantly. Deducting the expansion of the fused quartz bar from these dial readings gave the corrections which should be deducted from dial readings in later tests to obtain the expansion of specimens investigated. These corrections are shown by solid portion of curve *C*, figure 3. Appying these corrections to the readings obtained with the graphite bar (curve *A*) gave the values for the expansion of the bar itself below 1,000° C. which are shown by the solid portion of curve *B*, figure 3.[4]

Curve *A* showed no irregularities between 1,000° and 1,800° C.; consequently it is logical to assume that neither curve *B* nor *C* above 1,000° C. should show irregularities. Moreover, as the curvature of curve *A* in the temperature range 500 to 1,800° C. and also that of curve *B* in the range 500° to 1,000° C. were approximately constant, it seemed reasonable to assume that the curvature of curve B would remain approximately constant from 500° to 1,800° C. Accordingly curve *B* was extended from 1,000° to 1,800° C. based on that assumption. The calibration or correction curve *C* was then extrapolated to 1,800° C. by taking the difference between curves *A* and *B* at 100° C. intervals in the range 1,000° to 1,800° C.

A further precaution was taken in that the furnace was calibrated to 1,000° C. with the specimen of fused quartz preceding practically every test of a material.

IV. METHOD OF TESTING

Linear thermal expansion measurements were made of the same specimen, of each of the materials, in steps as follows: (1) Up to 1,000° C. at approximately 100° C. intervals, using either the entire specimen and the apparatus described in the Second Progress Report of the Sagger Investigation[5] or chips of the specimen and the interferometer.[6] (2) In the induction furnace to some temperature above 1,000° C., but in no case above 1,800° C. (3) The procedure described under (1) was repeated with the specimen taken from the induction furnace. (4) After the measurements described under (3) were completed, the specimen was again tested in the induction furnace at the higher temperatures. There were two reasons for this procedure, (*a*) the time for making the tests in the induction furnace could be considerably lessened because only a few observations below 1,000° C. would be necessary, and (*b*) an error in the set-up of the specimen in the induction furnace would be quickly detected because the data obtained below 1,000° C. with the induction furnace should generally

[4] The values obtained checked the expansion of the graphite bar determined over the range 20° to 500° C. by means of an apparatus described elsewhere. R. F. Geller and R. A. Heindl, II. Progress Report on Investigation of Sagger Clays—Some Observations as to the Significance of Their Thermal Expansions, J.Am.Cer.Soc., vol. 9, no. 9, pp. 555-575, 1926.

[5] See footnote 4.

[6] C. G. Peters and C. H. Cragoe, B.S.Sci. Paper No. 393, and G. E. Merritt, B.S.Sci. Paper No. 485. The specimens from which the chips were cut did not always receive the corrseponding heat treatment to 1,000° C.

agree with those previously obtained in the furnaces operating only to 1,000° C.

Temperatures in the induction furnace were measured with the thermocouples up to approximately 1,500 C., following which the couples were removed and the temperatures observed with an optical pyrometer. In testing with the induction furnace, temperature readings to and including 1,000° C. were taken at approximately 250° C. intervals, thereafter at approximately 100° C. intervals. It was always the practice to check the optical pyrometer against the thermocouples before their removal. Temperatures were maintained for approximately 15 minutes before the final micrometer dial readings were recorded for each temperature up to 1,500° C.; thereafter no effort was made to stop at any specific temperature. However, an attempt was made to regulate the power input so that readings were taken only while the furnace temperature was increasing at a comparatively slow rate or not at all.

The maximum temperature at which expansion or contraction movements were observed depended on the refractoriness of the material, but in no case did it exceed 1,800° C.

In most instances observations at temperatures above 1,000° C. were made of each specimen twice, namely, (1) after its preparation, and (2) after its initial test in the induction furnace. This procedure afforded the means of determining whether any changes had occurred in the expansions of the materials as a result of having been heated in the induction furnace at temperatures considerably higher than those at which they had been initially heated.

V. RESULTS

1. LINEAR THERMAL EXPANSION

Linear thermal expansion or contraction values for all the materials are shown in the curves given in figures 4 to 9, inclusive. Observations (filled in circles) with the induction furnace below 1,000° C. are not given in figures 5 to 9 because they very nearly coincided with the observations (clear circles) obtained in the furnaces operated only to 1,000° C.

Pertinent data are summarized in table 1. A detailed discussion of the data, therefore, is not necessary. The data obtained up to 1,000° C. have an accuracy of ±2½ percent [7] and it is estimated those above 1,000° C. have an accuracy of ±5 percent. The curves give the following information relative to expansion of the materials: (*a*) Type, whether uniform or nonuniform, (*b*) the effect, if any, of reheating, and (*c*) the temperature at which bloating or contraction begins. Table 1 gives the average coefficients of expansion for various temperature ranges.

[7] See footnote 4, p. 719, for reference to report giving calibration of apparatus.

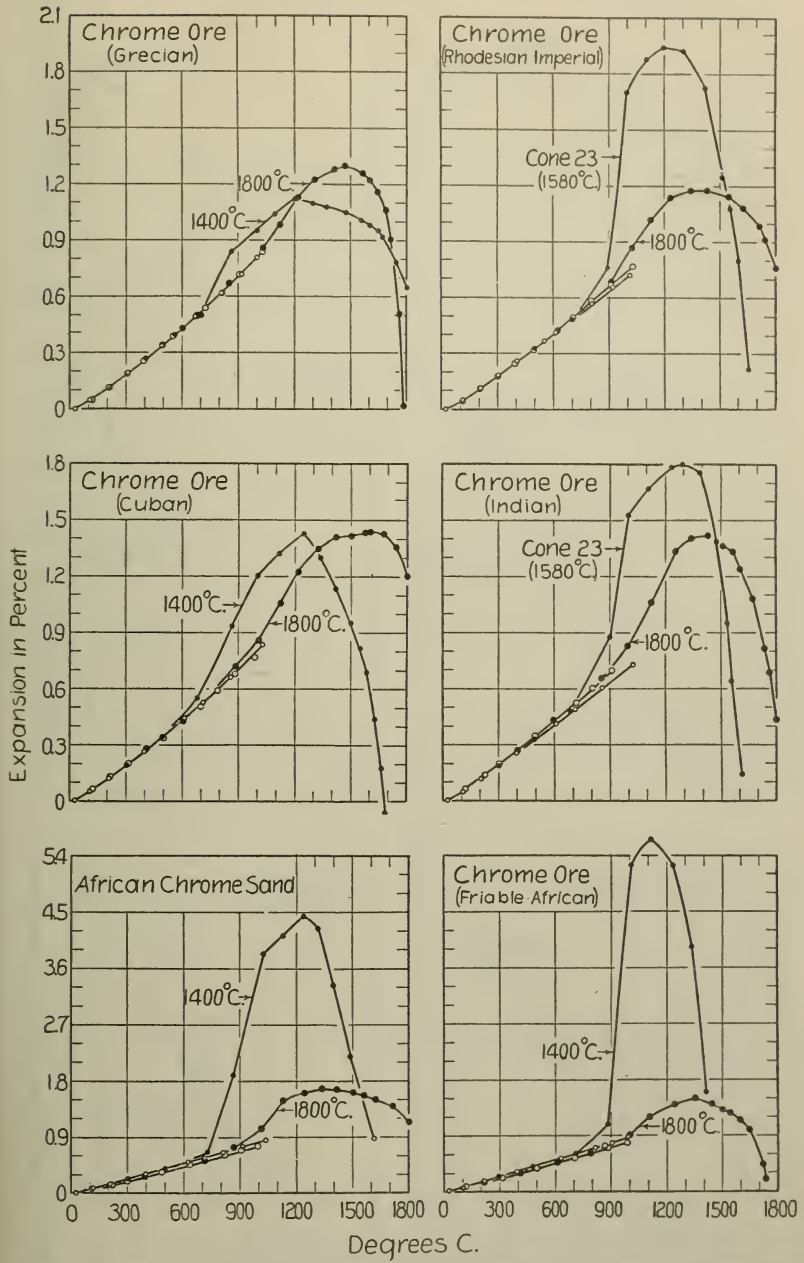

FIGURE 4.—*Thermal expansion curves for five chrome ores and a chrome sand.*

The temperatures adjacent to the curves indicate the maximum heat treatment the specimen had received previous to testing. The filled in circles on the curves represent the values obtained in a reducing atmosphere and the clear circles those obtained in an oxidizing atmosphere.

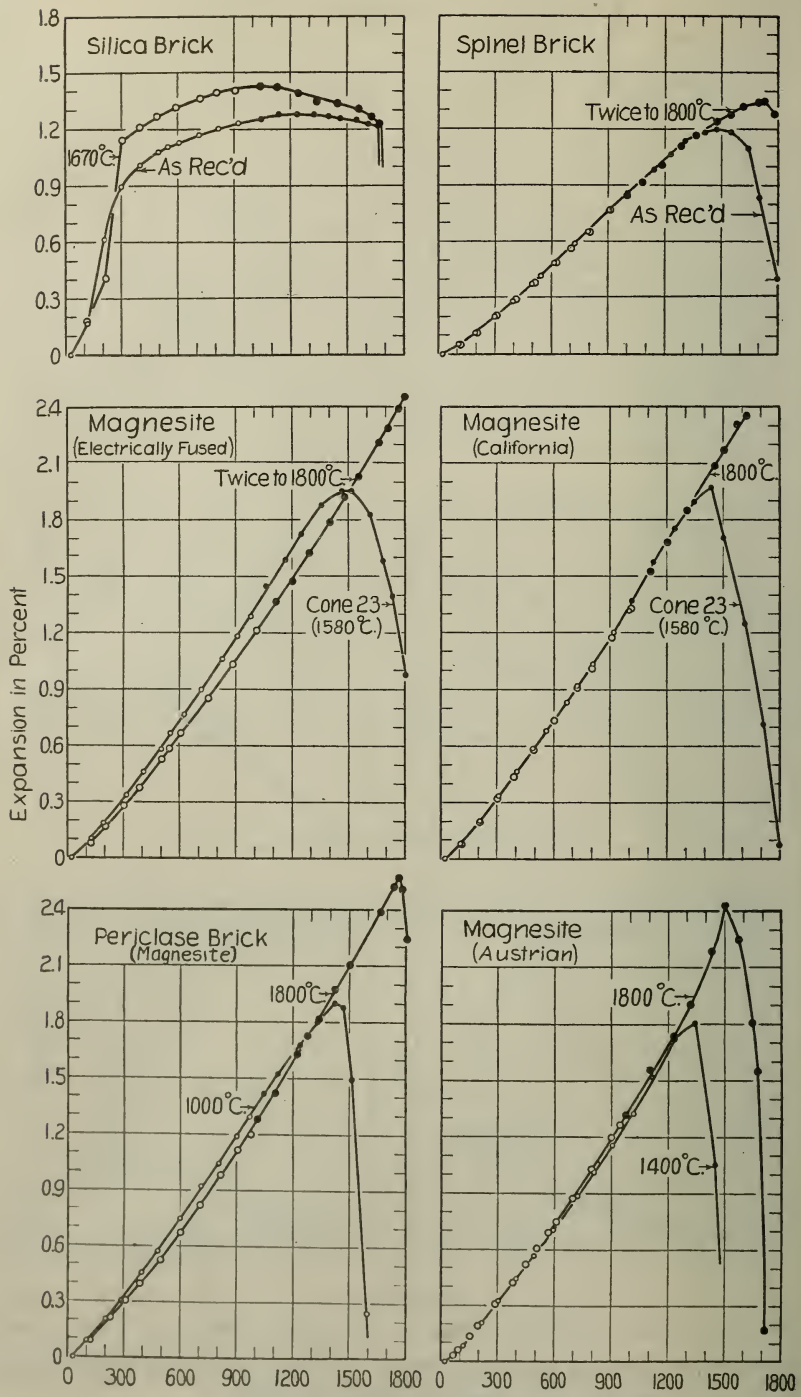

FIGURE 5.—*Thermal expansion curves for a silica brick, a spinel brick, and four magnesites.*

The temperatures adjacent to the curves indicate the maximum heat treatment the specimen had received previous to testing. (For additional details, see p. 721.)

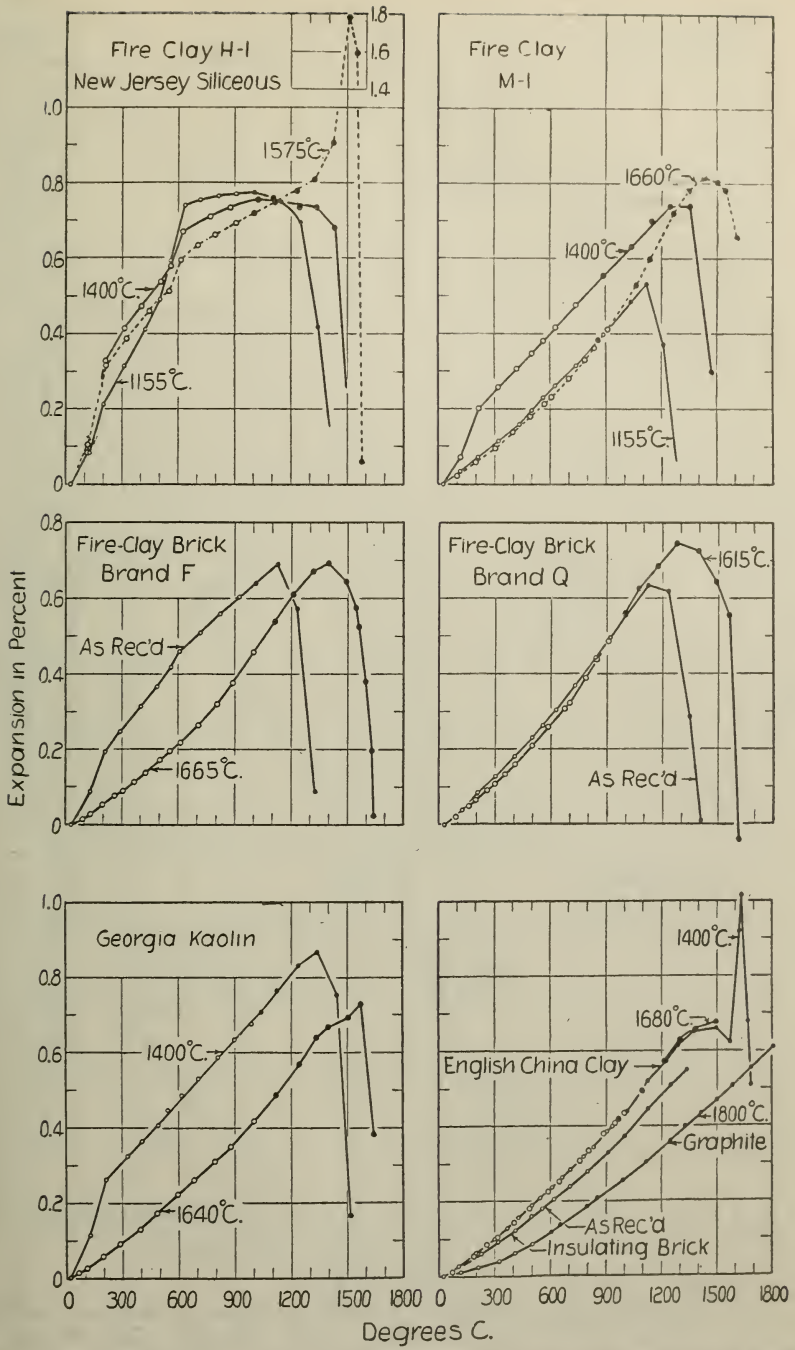

FIGURE 6.—*Thermal expansion curves for two types of fire clays and fire-clay bricks, a Georgia kaolin, an English china clay, insulating brick, and graphite.*

The temperatures adjacent to the curves indicate the heat treatment the specimen had received previous to testing. (For additonal details, see p. 721.)

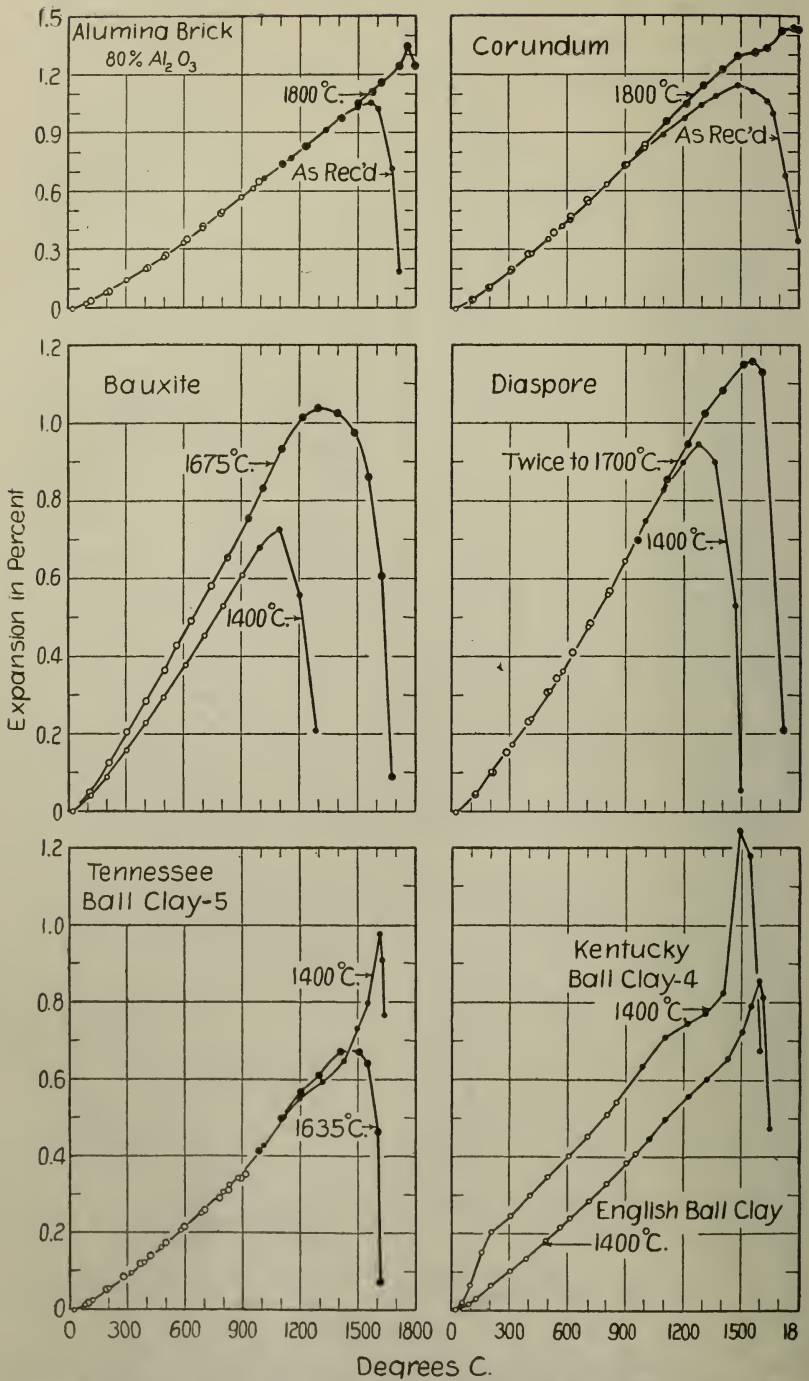

FIGURE 7.—*Thermal expansion curves for a high alumina fire brick, corundum, bauxite, diaspore, and Tennessee, Kentucky, and English ball clays.*

The temperatures adjacent to the curves indicate the heat treatment the specimen had received previous to the testing. (For additional details, see p. 721.)

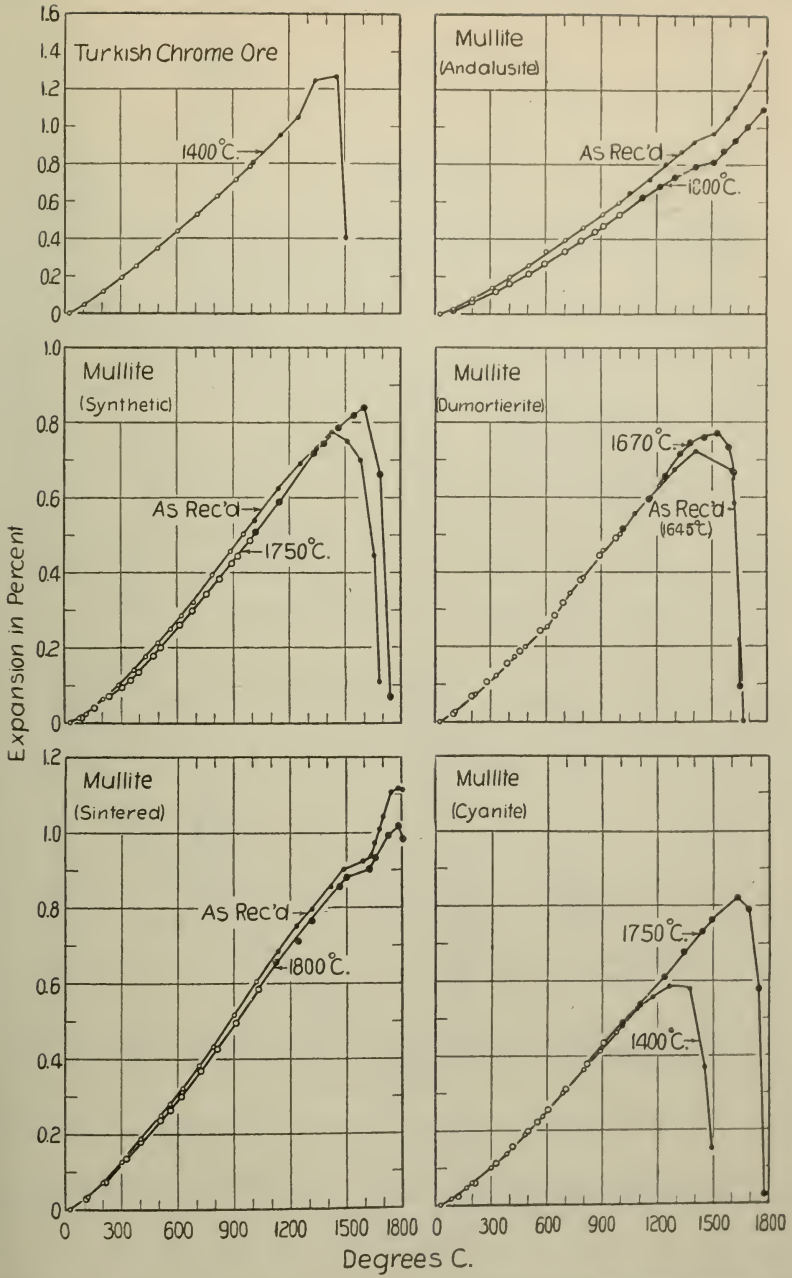

FIGURE 8.—*Thermal expansion curves for five mullites from different sources, and a chrome ore.*

The temperatures adjacent to the curves indicate the heat treatment the specimen had received previous testing. (For additional details, see p. 721.)

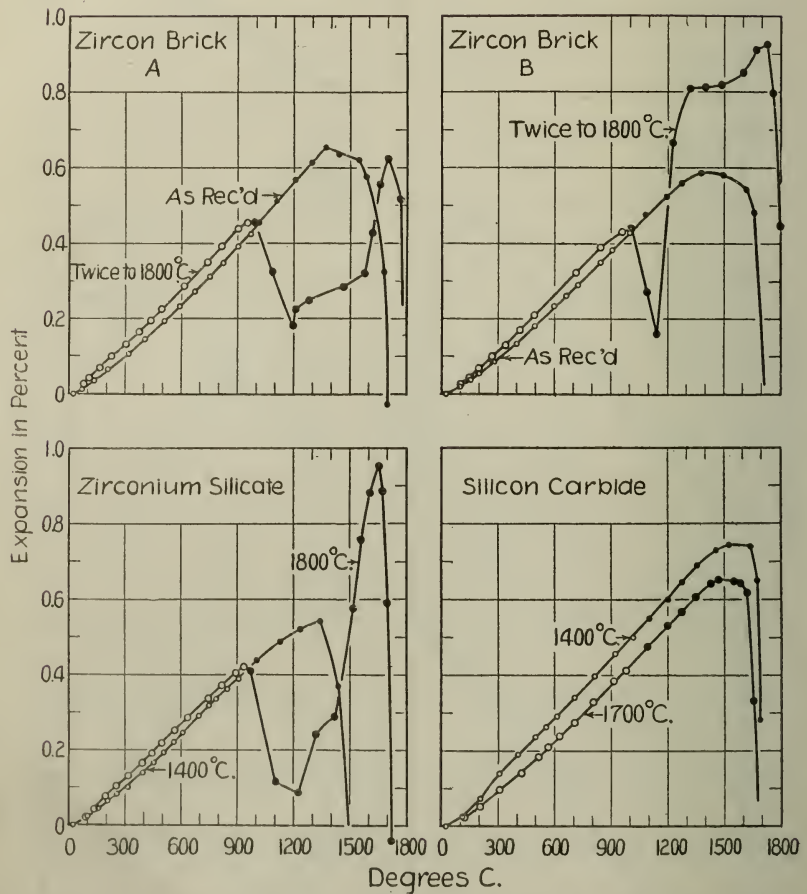

FIGURE 9.—*Thermal expansion curves for two zircon bricks, zirconium silicate, and silicon carbide.*

The temperatures adjacent to the curves indicate the heat treatment the specimen had received previous to testing. (For additional details, see p. 721.)

TABLE 1.—*Average coefficients of linear thermal expansion of refractories after different heat treatments*

Material	Chemical analyses [1]									Original heat treatment [2]	Maximum temperature reached in induction furnace		Linear thermal expansion in—						Total gain (+) or loss (−) after completion of tests, in—		Extent of deformation at end of tests
													Induction furnace				Resistance wire furnace [5]				
													First test		Second test		Average coefficient of expansion between 20° and 1,000° C.—				
	Ignition loss	SiO_2	FeO	Fe_2O_3	Al_2O_3	TiO_2	CaO	Cr_2O_3	MgO		First test	Second test	Temperature range [3]	Average coefficient of expansion [4]	Temperature range [3]	Average coefficient of expansion [4]	Before first test in induction furnace	After first test in induction furnace	Weight	Length	
	Per-cent	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*°C.*	*°C.*	*°C.*	*°C.*	$\times 10^{-6}$	*°C.*	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	*Per-cent*	*Per-cent*	
African chrome sand		4.5						51.3		1,400	1,800	1,800	20–700 700–1,000 1,000–1,235	8.1 108.0 28.0	20–700 700–1,000 1,000–1,125 1,125–1,475	7.5 15.4 40.0 8.6	8.2	7.2	−5.3	−3.3	Slight.
Cuban chrome ore	1.3	2.4	15.1		29.7			34.2	16.5	1,400	1,800	1,800	20–700 700–1,000 1,000–1,250	7.6 20.5 8.9	20–700 700–1,000 1,000–1,215 1,215–1,600	7.4 10.9 17.0 5.7	8.3	7.9	−3.7	−2.3	Do.
Grecian chrome ore	1.1	3.8	13.3		24.8			40.8	15.3	1,400	1,800	1,800	20–700 700–865 865–1,200	7.4 21.0 8.3	20–700 700–1,030 1,030–1,300 1,300–1,470	7.6 10.2 13.3 4.8	8.2	8.2	−3.1	−4.3	Do.

[1] The analyses for the chrome ores, magnesites, 80 percent alumina brick, corundum, and bauxite were furnished by the companies which supplied the materials; the remainder of those given were made at the Bureau of Standards by J. F. Klekotka and F. W. Glaze.

[2] Materials marked "As received" were tested without preliminary heating in the laboratory furnaces, since they have been heated in commercial furnaces previous to their receipt.

[3] Shrinkage occurred above the highest temperatures given in this column except in the case of the California magnesite (second test).

[4] The negative sign preceding a value indicates contraction.

[5] Description of furnace and method given in J. Am. Ceramic Soc. vol. 9 (9), pp. 556-574, 1926.

TABLE 1.—*Average coefficients of linear thermal expansion of refractories after different heat treatments*—Continued

Material	Chemical analyses: Ignition loss	SiO_2	FeO	Fe_2O_3	Al_2O_3	TiO_2	CaO	Cr_2O_3	MgO	Original heat treatment	Maximum temperature reached in induction furnace: First test	Second test	Linear thermal expansion in— Induction furnace: First test: Temperature range	First test: Average coefficient of expansion	Second test: Temperature range	Second test: Average coefficient of expansion	Resistance wire furnace: Average coefficient of expansion between 20° and 1,000° C.— Before first test in induction furnace	After first test in induction furnace	Total gain (+) or loss (−) after completion of tests, in— Weight	Length	Extent of deformation at end of tests
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	°C.	°C.	°C.	°C.	$\times 10^{-6}$	°C.	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	*Percent*	*Percent*	
Friable African chrome ore.	2.0	5.6	15.4	-----	13.3	-----	-----	49.0	13.7	1,400	1,800	1,740	20–700	8.4	20–700	7.5	8.8	7.8	−10.3	−4.9	Slight.
													700–900	25.0	700–1,000	12.4					
													900–1,115	199.0	1,000–1,350	16.7					
Imperial chrome ore, Rhodesian.	2.1	5.2	13.4	-----	15.8	-----	-----	50.4	12.1	Cone 23 (1,580)	1,800	1,800	20–700	7.1	20–700	7.2	7.2	7.6	−3.1	−3.2	None.
													700–900	14.0	700–900	10.7					
													900–1,000	93.0	900–1,230	13.9					
													1,000–1,200	12.0	1,230–1,430	2.2					
Indian chrome ore	1.5	5.5	21.6	-----	13.2	-----	-----	47.9	9.4	Cone 23 (1,580)	1,800	1,800	20–700	7.0	20–900	7.9	7.2	8.1	−3.1	−4.9	Very little, if any.
													700–900	18.6	900–1,000	13.4					
													900–1,000	65.0	1,000–1,250	19.5					
													1,000–1,300	9.4	1,250–1,430	4.7					
Turkish chrome ore	3.4	9.4	13.4	-----	15.6	-----	-----	40.0	16.8	1,400	1,555	------	20–1,340	9.4	----------	------	8.1	------	−5.9	−13.6	Almost sheared.
Austrian magnesite	-----	3.9	-----	8.2	2.2	-----	2.5	-----	83.3	1,400	1,800	1,800	20–1,340	13.7	20–1,500	16.4	13.3	13.7	+2.3	−14.3	Great.
California magnesite	-----	4.4	-----	5.8	2.3	-----	3.0	-----	84.2	Cone 23 (1,580)	1,800	1,630	20–1,440	13.9	20–1,630	14.6	13.6	13.6	+.4	−2.3	Slight.
Electrically fused magnesite.	-----	-----	-----	-----	-----	-----	-----	-----	-----	Cone 23 (1,580)	1,800	1,800	20–1,355	14.0	20–1,800	13.8	13.4	13.4	+3.4	−3.9	Do.

Periclase brick, California.										As received and 1,000° C.	1,800	1,800	20–1,420	13.6	20–1,765	14.7	13.7	12.9		−4.6	Very little, if any.
Spinel											1,800	1,800	20–1,410	8.5	20–1,730	7.8	8.5	8.5	+1.0	−1.7	Do.
Fire clay brick "Q"[6]	.2	47.1		1.4	46.8	2.5	1.2		.5	As received	1,615	1,630	20–1,120	5.8	20–1,280	5.9	5.7	5.5	−.9	−5.0	Moderate.
Fire clay brick "F"[6]	.3	59.6		1.7	34.5	2.0	.5		.6		1,665	1,660	20–1,130	6.2	20–1,395	5.1	6.3	4.7	−.3	−5.6	Slight.
Fire clay "H-1"[6]	7.4	70.3		.9	19.5	2.0	.2		.03		1,155	1,575	1,585	20–630 630–1,000 1,000–1,250	12.1 1.1 −3.3	20–610 610–1,320 1,320–1,510	10.1 3.0 53.0			−.5	−14.4	Do.
											1,400	1,585		20–610 610–1,020 1,020–1,425	11.4 2.1 −1.8							
Fire clay "M-1"[6]	13.7	44.6		1.1	38.2	2.2	.1		.1		1,155 1,400	1,660 1,670	1,600	20–1,120 20–200 200–1,250	4.9 10.9 5.2	20–1,430	5.8	4.8	5.0	−.6	−10.8	Very little, if any.
Kentucky ball clay no. 4.	12.5	51.9		.8	31.2	1.5	.3				1,400	1,600		20–200 200–1,400 1,400–1,490	10.8 5.2 47.2			6.1		−.1	−.3	Great (bloated).
Tennessee ball clay no. 5.	12.1	52.2		.8	31.2	1.1	.2				1,400	1,635	1,620	20–1,425	4.6	20–1,410	4.8	4.3	4.3	−.1	−.1	Slight (slightly bloated).
English ball clay "Dorset."	17.2	44.4		1.3	33.6	.8	.3		.5		1,400	1,650		20–1,430	4.6			4.4		−.5	−.3	Do.
Georgia kaolin[7]	13.7	45.3		.3	39.1	1.5	.1		.04		1,400	1,640	1,630	20–210 210–1,335	14.0 5.4	20–1,570	4.7		4.4	−.9	−2.0	Very little (bloated).
English china clay[7]	12.4	47.0		1.0	37.7	.2	.2		.2		1,400	1,680	1,680	20–1,375	4.8	20–1,375	4.8	4.4	4.4	−.03	−.6	Do.
Firebrick "80 percent alumina."		15.0		1.9	80.5				.6	As received (cone 18–1485).	1,800	1,800	20–1,570	6.8	20–1,760	8.0	6.7	6.6	+.7	−3.9	None.
Corundum, artificial		1.9		.5	95.5		1.9			As received (cone 16–1425).	1,800	1,800	20–1,485	7.8	20–1,800	8.1	8.5	8.4	−.9	+.2	Do.
Diaspore											1,400	1,700	1,720	20–1,275	7.5	20–1,550	7.6	7.8	7.7	−.1	−17.4	Slight.
Bauxite (calcined)	.7	4.7		9.7	81.7	3.2					1,400	1,675	1,690	20–1,100	6.7	20–1,300	8.1	6.8	8.2	−2.3	−15.4	Great.
Mullite (Dumortierite).										As received cone 29½–1645).	1,670	1,730		20–1,410	5.2	20–1,530	5.1	5.2	5.2		−1.1	Very little, if any.
Mullite (Andalusite)										As received (cone 16–1450).	1,800	1,800	20–1,400 1,400–1,510 1,510–1,785	6.7 4.3 15.9	20–1,410 1,410–1,510 1,510–1,780	5.7 2.3 10.7	6.2	5.3		+.8	None (bloated).
Mullite (sintered)										As received	1,800	1,800	20–1,475 1,475–1,585 1,585–1,760	6.2 2.0 10.8	20–1,495 1,495–1,620 1,620–1,780	6.0 1.8 7.3	6.0	5.7	−.5	+.2	None.
Mullite (Indian cyanite).		36.2		.04	63.4	.7	.2				1,400	1,750	1,800	20–1,260	4.7	20–1,630	5.1	4.9	5.0			Slight.
Mullite (synthetic)										As received	1,750	1,735	20–1,430	5.5	20–1,600	5.3	5.4	5.0		−5.6	Do.

[6] These materials are referred to in Bureau of Standards Research Paper No. 114.
[7] Additional information relative to these materials may be found in Bureau of Standards Research Paper No. 410.

TABLE 1.—*Average coefficients of linear thermal expansion of refractories after different heat treatments*—Continued

Material	Chemical analyses: Ignition loss	SiO_2	FeO	Fe_2O_3	Al_2O_3	TiO_2	CaO	Cr_2O_3	MgO	Original heat treatment	Maximum temperature reached in induction furnace: First test	Second test	Linear thermal expansion in— Induction furnace: First test: Temperature range	First test: Average coefficient of expansion	Second test: Temperature range	Second test: Average coefficient of expansion	Resistance wire furnace: Average coefficient of expansion between 20° and 1,000° C.: Before first test in induction furnace	After first test in induction furnace	Total gain (+) or loss (−) after completion of tests, in —: Weight	Length	Extent of deformation at end of tests
	Per-cent	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	°C.	°C.	°C.	°C.	$\times 10^{-6}$	°C.	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	*Per-cent*	*Per-cent*	
Zircon brick "A"										As received (1,700).	1,800	1,800	20–1,370	4.8	20– 900 900–1,200 1,200–1,615 1,615–1,715	5.0 −8.7 5.7 20.1	4.5	[8] 5.0	0	−5.6	Slight.
Zircon brick "B"										As received	1,800	1,800	20–1,375	4.3	20– 900 900–1,140 1,140–1,315 1,315–1,725	5.7 −14.3 36.5 2.9	4.4	[8] 5.7	−.6	+.2	Do.
Zirconium silicate (98 percent zircon)										1,400	1,800	1,800	20–1,340	4.1	20– 930 930–1,225 1,225–1,655	4.6 −11.4 20.0	4.4	[8] 4.6	−.4	−10.1	Do.
Silicon carbide										1,400	1,700	1,690	20–1,525	4.9	20–1,470	4.5	5.2	4.3	------	1.1	Sheared.
Silica brick										As received	1,670	1,670	20– 300 300–1,135 1,135–1,665	32.6 4.6 −1.1	20– 300 300–1,040 1,040–1,670	41.0 3.9 −3.3	------	------	−.3	−.04	None (very friable).
Insulating brick										As received	1,330	------	20–1,330	4.2	------	------	3.9	------	−.1	−.2	None.

[8] Average coefficient of expansion between 20° and 900° C.

The following generalizations may be made when comparing the different materials studied:

1. A comparison of the results obtained in oxidizing and reducing atmospheres showed that the expansions of the chrome ores were affected more by the reducing atmosphere than the other materials.
2. The magnesites showed the greatest total expansion of those materials which had nearly a uniform rate of expansion.
3. The expansion curves of the zircons in the second tests showed greater irregularities above 950° C. than the curves of the other materials. This was probably due to the decomposition of zirconium silicate into zirconium oxide and glass.
4. All materials showed some change in total expansion in the second test when compared with that obtained in the first test although in some instances the difference is small.
5. Nearly all the materials decreased in weight and length during the tests. The loss in weight was due to volatilization of one or more of the constituents. Such volatilized material condensed or sublimed in the upper tube of the set-up. In most cases this material was probably silica. Crystalline growths appeared on some of the specimens. At the end of the tests all chrome ores were covered with beads of metal, probably an alloy of iron and chromium.

2. PETROGRAPHIC ANALYSES [8]

Petrographic analyses were made on chips taken from the specimens of the materials before and after the various heat treatments in order to obtain information relative to the effect of the various heat treatments on their mineralogical constitution. The analyses follow:

AFRICAN CHROME SAND

As received.—Largely chromite. Grains mostly transparent. Index of refraction 2.07. Some hematite possibly present. Nonmagnetic.

Heated at 1,400° C.—Three major constituents present: (*a*) Red colored chromite, index 2.07, isotropic; (*b*) sharply delimited areas of opaque material in the chromite grains; (*c*) colorless, transparent grains, index slightly lower than 1.50.

Heated to 1,800° C.—Material has vitreous luster. Principal constituents present: (*a*) Chromite; (*b*) opaque material, less abundant than in previous samples; (*c*) interstitial material in spherulitic or needle-like forms, index about 1.65, intimately associated with an isotropic material of lower index, probably glass.

CUBAN CHROME ORE

As received.—Largely transparent chromite with some serpentine. Very little opaque material present.

Heated at 1,400° C.—Largely chromite with a few needle-like birefringent crystals.

Heated to 1,800° C.—Chromite changed very little. Many grains having the red color and high index of chromite show a slight double refraction, perhaps due to strain.

GRECIAN CHROME ORE

As received.—Largely brown transparent chromite with a small amount of serpentine and a few grains of opaque material.

Heated at 1,400° C.—Birefringent material, index higher than 1.70, and weak pleochroism, associated with chromite perhaps a pyroxene.

Heated to 1,800° C.—Chromite grains lighter in color. Opaque material present in distinct grains with sharp boundaries.

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[8] Made by H. Insley.

FRIABLE AFRICAN CHROME ORE

As received.—Composed of (*a*) chromite, (*b*) much opaque material, (*c*) small aggregates of red colored particles having a high index and high birefringence (possibly hematite), (*d*) small amount of quartz.

Heated at 1,400° C.—Material not greatly different from that not heated.

Heated to 1,800° C.—Chromite grains lighter in color. Less opaque material present than originally. Occurs as distinct, rounded grains. Possibly some pyroxene and glass or cristobalite present.

RHODESIAN IMPERIAL CHROME ORE

As received and after heating at cone 23 (1,580° C.) and 1,800 °C.—None of the samples differ greatly. In addition to the chromite, a small amount of low index glass and opaque material, possibly hematite, present.

INDIAN CHROME ORE

As received.—Largely chromite, transparent and brown in thin pieces, and an opaque material.

Heated to cone 23 (1,580° C.).—Not greatly different from the "as received" material except the opaque grains are rounded and have sharp boundaries against the chromite. Needlelike inclusions in chromite may be rutile.

Heated to 1,800° C.—Chromite grains slightly lighter in color. Opaque grains not greatly changed. Crystalline grains of birefringent material, index about 1.70, have appeared in appreciable quantities.

TURKISH CHROME ORE

As received.—Largely deep red chromite grains and serpentine with a small amount of pyroxene.

Heated at 1,400° C.—Less opaque material. A spherulitic or fibrous crystalline material with a low birefringence and index of about 1.70, probably a pyroxene, was found to be intimately associated with and surrounded by a low index, isotropic material, probably glass.

Heated to 1,555° C.—Composed of: (*a*) Chromite usually brown in color, (*b*) high index almost opaque material which may be a spinel like hercynite, (*c*) birefringent grains (index 1.65–1.70) probably pyroxene, (*d*) lower index material, probably glass.

AUSTRIAN MAGNESITE

As received (calcined).—Material very heterogeneous. Isotropic material, periclase, index of refraction ranging from 1.735 to 1.76 composed approximately 85 percent of sample. The chief impurities are: (*a*) Forsterite, (*b*) aggregates of what is probably hematite, and (*c*) a small amount of low index material, probably glass.

Heated to 1,800° C.—Periclase grains large and rounded. Red, birefringent material (hematite) is almost absent. The grains of forsterite are large and very nearly pure $2MgO.SiO_2$.

CALIFORNIA MAGNESITE

As received (calcined).—Composed of: (*a*) More than 90 percent periclase, index 1.735, (*b*) forsterite as interstitial material.

Heated to cone 23 (1,580) and 1,800° C.—Periclase clear and colorless, either in rounded grains or faceted crystals. Forsterite crystals increased in size. Opaque material occurs as spherical inclusions in the periclase.

ELECTRICALLY FUSED MAGNESITE

As received, heated to cone 23 (1,580° C.) and 1,800° C.—Composed of: (*a*) Over 90 percent periclase, (*b*) less than 1 percent glass and, (*c*) forsterite. All constituents colorless. No noticeable change due to different heat treatments.

PERICLASE BRICK (CALIFORNIA)

As received.—Principally composed of periclase, small amounts of forsterite and probably glass, and a few scattered grains of what may be a spinel.

Heated to 1,800° C.—The size of the periclase grains and crystals of forsterite increased, otherwise little change from the "as received" material.

SPINEL BRICK

As received.—Composed of over 90 percent spinel (index 1.72). A small amount of glass and a crystalline birefractory material (probably clinoenstatite or forsterite) distributed along the boundaries of the spinel grains.

Heated to 1,800° C.—The spinel and glass grains increased in size and the birefractory material disappeared.

BRICK, FLINT CLAY, "BRAND Q"

As received.—Composed largely of glass and very small, poorly developed mullite crystals, with a few grains of quartz.

Heated to 1,630° C.—Mullite crystals increased in size, quartz dissolved. Apparently more glass present than mullite.

BRICK, SEMISILICEOUS, "BRAND F"

As received.—Composed principally of aggregates with a mean index of refraction of 1.56. These aggregates are probably glass or cristobalite and feebly developed needles of mullite. Some grains of quartz are visible.

Heated to 1,660° C.—Mullite needles well developed. Quartz completely disappeared. More glass (index 1.50) than mullite present.

FIRECLAY, HIGHLY SILICEOUS, "H-1"

Heated at 1,155° C.—Principally glass, quartz grains surrounded with cristobalite and poorly developed needles of mullite.

Heated to 1,585° C.—Not greatly different from preceding material.

FIRECLAY, M-1

Heated at 1,155° C.—Amorphous material (mean index 1.56) containing fibrous material with a lower index. Mullite not identifiable.

Heated to 1,670° C.—Small irregular needles of mullite and an interstitial material not identifiable.

KENTUCKY BALL CLAY

Raw.—Largely (*a*) platy clay mineral, often in vermicular growths, probably kaolinite; (*b*) quartz; (*c*) amorphous material (probably a clay mineral) with index about 1.55; (*d*) amorphous brown material; (*e*) a little rutile.

Heated at 1,400° C.—Largely poorly developed mullite needles embedded in a lower index matrix.

Heated to 1,600° C.—Principally mullite and glass.

TENNESSEE BALL CLAY

Raw.—Composition similar to that of the above Kentucky ball clay.

Heated at 1,400° C.—Wholly mullite and glass with apparently more glass than mullite.

Heated to 1,635° C.—Mullite crystals well developed with glass more abundant than mullite.

ENGLISH BALL CLAY

Raw.—Material extremely fine grained. Largely amorphous material and a platy clay mineral. Considerable organic matter, a little rutile, quartz, and muscovite also present.

Heated at 1,400° C.—A mass of poorly developed mullite crystals in a matrix of glass.

Heated at 1,650° C.—Considerably more glass than mullite present. Mullite grains show much evidence of solution.

GEORGIA KAOLIN

Raw.—Contains approximately 2 percent mica and less than 1 percent quartz. Remainder kaolinte.

Heated at 1,400° C.—Principally extremely small needles of mullite and an interstitial material.

Heated to 1,660° C.—Composed wholly of well developed crystals of mullite and glass.

ENGLISH CHINA CLAY

Raw.—Contains from 9 to 11 percent of muscovite, less than 1 percent of quartz, and less than 2 percent of feldspar. Remainder kaolinte.

Heated at 1,400° C.—Almost wholly well-developed needles of mullite and glass with the latter in preponderance.

Heated to 1,680° C.—The amount of glass increased, indicating the solution of the mullite.

FIREBRICK, 80 PERCENT ALUMINA

As received.—Largely corundum and mullite. A small amount of glass and a few grains high index, high birefringent material (rutile or zircon).

Heated to 1,800° C.—Mullite and corundum present in about equal proportions. A small amount of glass still present, but the high index material previously noted gone.

CORUNDUM, ARTIFICIAL

As received.—Largely corundum with small amounts of glass and a high index, high birefringent material (possibly rutile).

Heated to 1,800° C.—Not much change from the "as received" material.

DIASPORE

Raw.—Mainly diaspore with a very small amount of fine-grained clay mineral.

Heated at 1,400° C.—Almost wholly corundum and glass.

Heated to 1,720° C.—Corundum grains much larger. A small amount of mullite occurs as interstitial material associated with glass.

BAUXITE

Heated to 1,400° C.—Principally corundum, with lesser amounts of mullite, glass, and a compound of what is probably Fe_2O_3 and TiO_2.

Heated to 1,690° C.—Not greatly changed from preceding sample except that the size of the mullite crystals increased and the crystals of corundum show evidence of reaction with the fluxes present.

MULLITE (DUMORTIERITE)

Heated to 1,730° C.—Wholly mullite and glass, the latter composing 30 percent or more of the sample.

MULLITE (ANDALUSITE)

Heated to 1,800° C.—Mullite, corundum, and glass. The amount of corundum estimated at approximately 15 percent and the glass from 5 to 10 percent.

MULLITE (SINTERED)

Heated to 1,800° C.—Mullite, corundum, and glass. The amount of corundum estimated at approximately 25 percent and the glass between 15 and 20 percent.

MULLITE (INDIAN CYANITE)

Heated to 1,800° C.—Glass and corundum and well-developed crystals of mullite. The corundum present estimated at less than 15 percent.

MULLITE (SYNTHETIC)

Heated to 1,750° C.—Principally mullite, with very small percentages of corundum and glass.

ZIRCON BRICK A

As received.—Coarse-grained zircon with a small amount of glass.

Heated to 1,800° C.—Two phases present: (*a*) Aggregates of zirconium oxide, (*b*) brownish glass. The zirconium oxide crystals were identified by X-ray diffraction pattern.

ZIRCON BRICK B AND ZIRCONIUM SILICATE

Similar to zircon brick A.

SILICON CARBIDE

As received and heated to 1,715° C.—Wholly pure silicon carbide.

SILICA BRICK

As received.—Largely tridymite with a lesser amount of cristobalite. A few remnants of quartz grains observed as well as an interstitial material, present to the extent of approximately 5 percent.

Heated to 1,670° C.—Almost wholly cristobalite with a small amount of isotropic interstitial material (glass).

INSULATING BRICK

As received.—Only minute crystals of mullite embedded in a greater proportion of matrix material, probably glass.

Heated to 1,330° C.—No pronounced change.

VI. SUMMARY

With four exceptions linear thermal expension measurements were made of each of 36 refractory materials using the specimen throughout the following cycle: (1) Room temperature to 1,000° C. in an oxidizing atmosphere, (2) room temperature to some temperature above, 1,000° C. but not exceeding 1,800° C., in a reducing atmosphere, (3) room temperature to 1,000° C. in an oxidizing atmosphere, (4) room temperature to some temperature above 1,000° C. but not exceeding 1,800° C., in a reducing atmosphere. This procedure gave information relative to the effect on the expansion of the materials (1) when tested in an oxidizing or reducing atmosphere, and (2) when heated at a temperature higher than that which they received preliminary to the first test.

The petrographic analyses give information relative to the constitution of the materials before and after the several heat treatments.

It was noted that:

1. No apparent differences in expansion were obtained from room temperature to 1,000° C. when the materials were tested in either a reducing or oxidizing atmosphere except in the case of chrome ores which showed an exceptionally high expansion between 700° and 1,000° C. under reducing conditions.

2. The magnesites showed the greatest total expansion of the materials which had nearly a uniform rate of expansion.

3. The expansion curves of the zircons in the second tests showed greater irregularities above 950° C. than the curves of the other materials. This was probably due to the decomposition of zirconium silicate into zirconium oxide and glass.

4. All materials showed some changes in total expansion in the second test when compared with that obtained in the first test although in some instances the difference is small.

5. Nearly all of the materials decreased in weight and length during the tests. The loss in weight was due to the volatilization of one or more of the constituents. Such volatilized material condensed or sublimed in the upper tube of the set-up. In most cases this material was probably silica. Crystalline growths appeared on some of the specimens. At the end of the tests all chrome ores were covered with beads of metal, probably an alloy of iron and chromium.

WASHINGTON, January 12, 1933.

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